# REPORT DOCUMENTATION PAGE

Nano/Micro Characterization

UU

a. REPORT

UU

16. SECURITY CLASSIFICATION OF:

b. ABSTRACT | c. THIS PAGE

UU

Form Approved OMB NO. 0704-0188

The public reporting burden for this collection of information is estimated to average 1 hour per response, including the time for reviewing instructions, searching existing data sources, gathering and maintaining the data needed, and completing and reviewing the collection of information. Send comments regarding this burden estimate or any other aspect of this collection of information, including suggesstions for reducing this burden, to Washington Headquarters Services, Directorate for Information Operations and Reports, 1215 Jefferson Davis Highway, Suite 1204, Arlington VA, 22202-4302. Respondents should be aware that notwithstanding any other provision of law, no person shall be subject to any oenalty for failing to comply with a collection of information if it does not display a currently valid OMB control number. PLEASE DO NOT RETURN YOUR FORM TO THE ABOVE ADDRESS.

1. REPORT DATE (DD-MM-YYYY)	2. REPORT TYPE		3. DATES COVERED (From - To)
24-06-2015	Final Report		12-Feb-2014 - 11-Feb-2015
4. TITLE AND SUBTITLE		5a. C	ONTRACT NUMBER
Final Report: Establishment of a Versa	<del>_</del>	W91	1NF-14-1-0090
Mechanical Characterization_NMMC_	_Facility	5b. G	RANT NUMBER
		5c. PF 1060	ROGRAM ELEMENT NUMBER 12
6. AUTHORS Kwang Kim		5d. PF	ROJECT NUMBER
		5e. TA	ASK NUMBER
		5f. W	ORK UNIT NUMBER
7. PERFORMING ORGANIZATION NAM	ES AND ADDRESSES		8. PERFORMING ORGANIZATION REPORT
University of Nevada - Las Vegas 4505 Maryland Parkway Box 454019 Las Vegas, NV 8915	54 -4019		NUMBER
9. SPONSORING/MONITORING AGENCY (ES)	Y NAME(S) AND ADDRESS		10. SPONSOR/MONITOR'S ACRONYM(S) ARO
U.S. Army Research Office P.O. Box 12211 Research Triangle Park, NC 27709-2211			11. SPONSOR/MONITOR'S REPORT NUMBER(S)
			64774-MS-REP.1
12. DISTRIBUTION AVAILIBILITY STAT			
Approved for Public Release; Distribution Un	limited		
13. SUPPLEMENTARY NOTES The views, opinions and/or findings contained of the Army position, policy or decision, unless			and should not contrued as an official Department
14. ABSTRACT			
many Department of Defense (DoD) a (UNLV), having diverse research interintegrated nano-analysis capability as in-situ investigation of materials of int	pplications. A group of reservests, intended to establish of well as a mechanical testing erest by looking into stress/	earcher our SEN g syster strain	M imaging capabilities by adding an m. The mechanical testing facility allows
15. SUBJECT TERMS			

17. LIMITATION OF

OF PAGES

ABSTRACT

UU

15. NUMBER 19a. NAME OF RESPONSIBLE PERSON

19b. TELEPHONE NUMBER

Kwang Kim

702-774-1419

# **Report Title**

Final Report: Establishment of a Versatile Nano\_Micro-Mechanical Characterization\_NMMC\_Facility

#### **ABSTRACT**

Nano/Micro-Mechanical characterization is one of the key elements for the development of material systems of many Department of Defense (DoD) applications. A group of researchers at University of Nevada, Las Vegas (UNLV), having diverse research interests, intended to establish our SEM imaging capabilities by adding an integrated nano-analysis capability as well as a mechanical testing system. The mechanical testing facility allows in-situ investigation of materials of interest by looking into stress/strain curves, shear, tensile, 3-point bend, Young's Modulus; simultaneously, allowing video-taping and generating other imaging reports. Furthermore, we added the capability of testing biological samples to make this SEM readily available for researchers who are in the field of biological sciences and engineering. In addition to the tabletop SEM, we also installed a Scanning ElectroChemical Workstation (SECW) that is advanced equipment for scanning probe electrochemistry designed for ultra-high resolution, non-contact, spatially resolved electrochemical measurements. This SECW helps UNLV students and researches to study through membranes, monitor biological activity, image chemical biosystems, and perform fuel cell materials, surface science, and corrosion science research.

Enter List of papers submitted or published that acknowledge ARO support from the start of the project to the date of this printing. List the papers, including journal references, in the following categories:

(a) Papers published in peer-reviewed journals (N/A for none)

Received Paper

TOTAL:

Number of Papers published in peer-reviewed journals:

(b) Papers published in non-peer-reviewed journals (N/A for none)

Received Paper

TOTAL:

Number of Papers published in non peer-reviewed journals:

(c) Presentations

Number of Pres	Number of Presentations: 0.00					
	Non Peer-Reviewed Conference Proceeding publications (other than abstracts):					
Received	<u>Paper</u>					
TOTAL:						
Number of Non	Peer-Reviewed Conference Proceeding publications (other than abstracts):					
	Peer-Reviewed Conference Proceeding publications (other than abstracts):					
Received	<u>Paper</u>					
TOTAL:						
Number of Peer	-Reviewed Conference Proceeding publications (other than abstracts):					
	(d) Manuscripts					
Received	<u>Paper</u>					
TOTAL:						

Number of Man	user ip is:			
		Books		
Received	<u>Book</u>			
TOTAL:				
Received	Book Chapter			
TOTAL:				
		Patents Submi	tted	
		Patents Awar	1.1	
		ratents Awar	ueu	
The PL Kwang k	Cim was awarded the 20	Awards 15 Nevada Board of Regents' Resea	urcher Award in March 2015	
The 11, It wang 1	thir was awarded the 20	15 110 rada Board of Regents Reser	nonor reward in major 2013.	
		Graduate Stud	ents	
NAME		PERCENT_SUPPORTED	Discipline	
Shelby N	elson	0.00		
Blake Na Sarah Tr		0.00 0.00		
Qi Shen	avid	0.00		
Tyler Sta	lbaum	0.00		
FTE Equ	ivalent:	0.00		
Total Nu		5		
	<del>-</del>	~		

## **Names of Post Doctorates**

NAME	PERCENT_SUPPORTED	
Viljar Palmre	0.00	
Taeseon Hwang	0.00	
Aupam Kumar	0.00	
FTE Equivalent:	0.00	
Total Number:	3	

### Names of Faculty Supported

<u>NAME</u>	PERCENT_SUPPORTED	National Academy Member
Kwang Kim	0.00	
FTE Equivalent:	0.00	
Total Number:	1	

# Names of Under Graduate students supported

NAME	PERCENT_SUPPORTED	Discipline
Blake Naccarato	0.00	Mechanical Engineering
Zakai Olsen	0.00	Mechanical Engineering
FTE Equivalent:	0.00	
Total Number:	2	

### **Student Metrics**

This section only applies to graduating undergraduates supported by this agreement in this reporting period

The number of undergraduates funded by this agreement who graduated during this period: ..... 1.00 The number of undergraduates funded by this agreement who graduated during this period with a degree in science, mathematics, engineering, or technology fields:..... 1.00

The number of undergraduates funded by your agreement who graduated during this period and will continue to pursue a graduate or Ph.D. degree in science, mathematics, engineering, or technology fields:..... 1.00

Number of graduating undergraduates who achieved a 3.5 GPA to 4.0 (4.0 max scale):..... 1.00 Number of graduating undergraduates funded by a DoD funded Center of Excellence grant for Education, Research and Engineering:..... 0.00

The number of undergraduates funded by your agreement who graduated during this period and intend to work for the Department of Defense ...... 0.00

The number of undergraduates funded by your agreement who graduated during this period and will receive scholarships or fellowships for further studies in science, mathematics, engineering or technology fields: ..... 1.00

## Names of Personnel receiving masters degrees

NAME		
Shelby Nelson		
Total Number:	1	

## Names of personnel receiving PHDs

<u>NAME</u>			
Total Number:			

	Names of other research staff
<u>NAME</u>	PERCENT_SUPPORTED
FTE Equivalent: Total Number:	

**Sub Contractors (DD882)** 

**Inventions (DD882)** 

# **Scientific Progress**

#### **Table of Contents**

- A. Establishment of a Versatile Nano/Micro-Mechanical Characterization Facility
- B. Table-Top SEM
- C. Scanning ElectroChemical Workstation (SECW)
- D. Other Equipment
- E. Example Images Taken at NMMC
- F. Work Plan
- G. Major Problem/Issues
- H. Project-related PublicationsAppendix –I

#### A. Establishment of a Versatile Nano/Micro-Mechanical Characterization Facility

Nano/Micro-Mechanical characterization is one of the key elements for the development of material systems of many Department of Defense (DoD) applications. A group of researchers at University of Nevada, Las Vegas (UNLV), having diverse research interests, intended to establish our SEM imaging capabilities by adding an integrated nano-analysis capability as well as a mechanical testing system. The mechanical testing facility allows in-situ investigation of materials of interest by looking into stress/strain curves, shear, tensile, 3-point bend, Young's Modulus; simultaneously, allowing video-taping and generating other imaging reports. Furthermore, we added the capability of testing biological samples to make this SEM readily available for researchers who are in the field of biological sciences and engineering. In addition to the tabletop SEM, we also installed a Scanning ElectroChemical Workstation (SECW) that is advanced equipment for scanning probe electrochemistry designed for ultra-high resolution, non-contact, spatially resolved electrochemical measurements. This SECW helps UNLV students and researches to study through membranes, monitor biological activity, image chemical biosystems, and perform fuel cell materials, surface science, and corrosion science research.

Establishment of such a versatile Nano/Micro-Mechanical Characterization (NMMC) facility greatly helped advance state-of-the art materials research at UNLV. The established NMMC facility is now being used in all the DoD-related research projects as well as other scientific research at the UNLV campus.

#### B. Table-Top SEM

A new tabletop SEM was purchased from Hitachi High Technologies America, Inc. (Model # TM3030, see Fig. 1). It is capable of generating high contrast and sharp images even at a high magnification. It is equipped with a low-vacuum mode owing to which no special sample preparation is necessary. Our SEM is integrated with an EDX unit to analyze the chemical components of materials under SEM in addition to a nano-/micro-scale mechanical testing system. The SEM can be used accelerating voltages of 5 kV, 10 kV, and 15 kV. Samples with diameters up to 70 mm and thicknesses up to 50 mm can be tested with the facility producing a magnification of about 15x–30,000x.

Additionally, the samples can be moved up to 35 mm in both X and Y directions. This table-top SEM is also capable of special stages like motorized stage, tilt rotate stage, and cooling stage (which is useful when imaging biological systems). All these features enable the SEM for advanced topographic imaging with a large depth of focus. One of the advantages arising out of the incorporation of mechanical testing feature in the SEM is that one can see the electron micrograph of the samples while tensile or compression testing is being performed. The SEM is appropriate for a wide range of research which includes materials, entomology, medicine, food, botany, cosmetics, semiconductor, textiles, and environment.

#### C. Scanning ElectroChemical Workstation (SECW)

In addition to the tabletop SEM, the UNLV's NMMC facility also installed a Scanning ElectroChemical Workstation (SECW) bought from Uniscan Instruments, Inc. (Model # M370, see Fig. 2). It is an advanced equipment for scanning probe electrochemistry designed for ultra-high resolution, non-contact, spatially resolved electrochemical measurements. It possesses a fast and precise positioning system with micro/nanometer resolution, along with a flexible data acquisition to select the configuration most suited for a particular experiment. The system is capable of being used in applications ranging from flow through membranes, monitoring biological activity, imaging of chemical biosystems (including immobilized enzymes), fuel cell materials research, and surface and corrosion science.

### D. Other Equipment

Other miscellaneous equipment including a critical point dryer and a sputter coater were purchased and installed in room SEB 1119 (Electron Microanalysis and Imaging Laboratory. See Fig. 3).

Sputtering System: We acquired a sputtering system from Denton Vacuum, Inc. (Model # DSK00V-0018) which is capable of depositing a large variety of coating materials (including iridium). The adjustable rotating and tilting stage ensures a highly uniform coating even on samples with highly irregular surfaces. It is capable of converting easily from metal sputter to carbon evaporation and is equipped with a turbo pump. The high resolution magnetron sputtering system is capable of depositing a conductive coating not only for scanning electron microscopy (SEM), but also for transmission electron microscopy (TEM) sample preparation.

Critical Point Dryer: Our critical point dryer was purchased from Electron Microscopy Sciences, Inc. (Model # EMS 850) which is capable of thermoelectric heating and adiabatic cooling with a temperature control +5°C on cooling and +35°C on heating. It avoids excess pressures or temperatures and ensures that the critical point is obtained. This obviates the need to rely on pressure relief valves to control pressure during the heating cycle. It is designed for use with CO2, after first replacing any water in the specimen by a series of dehydration steps.

E. Example Images Taken at NMMC

Below shows a variety of images taken by the table top SEM.

F. Work Plan

All the equipment was purchased and installed properly. They are operational.

G. Major Problem/Issues

None.

- H. Project-related Publications
- J.2 A New Ionic Polymer-Metal Composite Based on Nafion / poly(vinyl alcohol-co-ethylene) Blends T. Hwang, V. Palmre, J. Nam, D.-C. Lee, and K. J. Kim (submitted for review)
- J.1 A Comprehensive Physics-Based Model Encompassing Variable Surface Resistance and Underlying Physics of Ionic Polymer-Metal Composite Actuators Q. Shen, V. Palmre, T. Stalbaum, and K. J. Kim (submitted for review)

**Technology Transfer** 



# **Final Report**

Project Title: Establishment of a Versatile Nano/Micro-Mechanical Characterization Facility

Award Number: W911NF-14-1-0090

PIs Name: Kwang J. Kim<sup>1</sup> (PI) and 9 Co PIs<sup>2</sup>

Organization: University of Nevada, Las Vegas

Period: 12 February 2014 – 11 February 2015

Distribution: Dr. David, Stepp, Army Research Office

919-549-4329, email: david.m.stepp.civ@mail.mil

and

Dr. Thomas McKenna, Office of Naval Research

Phone: 703-696-4503, email: tom.mkenna@navy.mil

\_

South West Gas Professor of Energy and Matter, Mechanical Engineering Department, University of Nevada, Las Vegas, 4505 Maryland Parkway, Las Vegas, NV 89154-4027, Office: 702-774-1419 (<a href="mailto:kwang.kim@univ.edu">kwang.kim@univ.edu</a>)

Jaci Batista, Dept. of Civil and Environmental Eng., UNLV; Thomas Hartmann, Dept. of Mechanical Eng., UNLV; David W. Hatchett, Department of Chemistry, UNLV; Brendan O'Toole, Dept. of Mechanical Eng., UNLV; Laurel A. Raftery, School of Life Sciences, UNLV; Mohamed Trabia, Dept. of Mechanical Eng., UNLV; Woosoon Yim, Dept. of Mechanical Eng., UNLV; and Hui Zhao, Dept. of Mechanical Eng., UNLV

# **Table of Contents**

		Page
A.	Establishment of a Versatile Nano/Micro-Mechanical Characterization Facility	3
В.	Table-Top SEM	3
C.	Scanning ElectroChemical Workstation (SECW)	4
D.	Other Equipment	4
E.	Example Images Taken at NMMC	5
F.	Work Plan	5
G.	Major Problem/Issues	5
Н.	Project-related Publications	6
	Appendix –I	7

#### A. Establishment of a Versatile Nano/Micro-Mechanical Characterization Facility

Nano/Micro-Mechanical characterization is one of the key elements for the development of material systems of many Department of Defense (DoD) applications. A group of researchers at University of Nevada, Las Vegas (UNLV), having diverse research interests, intended to establish our SEM imaging capabilities by adding an integrated nano-analysis capability as well as a mechanical testing system. The mechanical testing facility allows *in-situ* investigation of materials of interest by looking into stress/strain curves, shear, tensile, 3-point bend, Young's Modulus; simultaneously, allowing videotaping and generating other imaging reports. Furthermore, we added the capability of testing biological samples to make this SEM readily available for researchers who are in the field of biological sciences and engineering. In addition to the tabletop SEM, we also installed a Scanning ElectroChemical Workstation (SECW) that is advanced equipment for scanning probe electrochemistry designed for ultra-high resolution, non-contact, spatially resolved electrochemical measurements. This SECW helps UNLV students and researches to study through membranes, monitor biological activity, image chemical biosystems, and perform fuel cell materials, surface science, and corrosion science research.

Establishment of such a versatile Nano/Micro-Mechanical Characterization (NMMC) facility greatly helped advance state-of-the art materials research at UNLV. The established NMMC facility is now being used in all the DoD-related research projects as well as other scientific research at the UNLV campus.

#### **B. Table-Top SEM**

A new tabletop SEM was purchased from Hitachi High Technologies America, Inc. (Model # TM3030, see Fig. 1). It is capable of generating high contrast and sharp images even at a high magnification. It is equipped with a low-vacuum mode owing to which no special sample preparation is necessary. Our SEM is integrated with an EDX unit to analyze the chemical components of materials under SEM in addition to a nano-/micro-scale mechanical testing system. The SEM can be used accelerating voltages of 5 kV, 10 kV, and 15 kV. Samples with diameters up to 70 mm and thicknesses up to 50 mm can be tested with the facility producing a magnification of about 15x-30,000x.



**Figure 1. Tabletop SEM** (installed in room SEB 2112A)

Additionally, the samples can be moved up to 35 mm in both X and Y directions. This table-top SEM is also capable of special stages like motorized stage, tilt rotate stage, and cooling stage (which is useful when imaging biological systems). All these features enable the SEM for advanced topographic imaging with a large depth of focus. One of the advantages arising out of the incorporation of mechanical testing feature in the SEM is that one can see the electron micrograph of the samples while tensile or compression testing is being performed. The SEM is appropriate for a wide range of research which includes materials, entomology, medicine, food, botany, cosmetics, semiconductor, textiles, and environment.

#### C. Scanning ElectroChemical Workstation (SECW)

In addition to the tabletop SEM, the UNLV's NMMC facility also installed a Scanning ElectroChemical Workstation (SECW) bought from Uniscan Instruments, Inc. (Model # M370, see Fig. 2). It is an advanced equipment for scanning probe electrochemistry designed for ultra-high resolution, non-contact, spatially resolved electrochemical measurements. It possesses a fast and precise positioning system with micro/nanometer resolution, along with a flexible data acquisition to select the configuration most suited for a particular experiment. The system is capable of being used in applications ranging from flow through membranes, monitoring biological activity, imaging of chemical biosystems (including immobilized enzymes), fuel cell materials research, and surface and corrosion science.

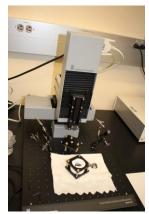


Figure 2. Scanning ElectroChemical
Workstation (SECW)
(installed in room SEB 2112A)



Figure 3. Sputtering System and Critical Point Dryer (installed in room SEB 1119)

## **D. Other Equipment**

Other miscellaneous equipment including a critical point dryer and a sputter coater were purchased and installed in room SEB 1119 (Electron Microanalysis and Imaging Laboratory. See Fig. 3).

<u>Sputtering System</u>: We acquired a sputtering system from Denton Vacuum, Inc. (Model # DSK00V-0018) which is capable of depositing a large variety of coating materials (including iridium). The adjustable rotating and tilting stage ensures a highly uniform coating even on samples with highly irregular surfaces. It is capable of converting easily from metal sputter to carbon evaporation and is equipped with a turbo pump. The high resolution magnetron sputtering system is capable of depositing a conductive coating not only for scanning electron microscopy (SEM), but also for transmission electron microscopy (TEM) sample preparation.

<u>Critical Point Dryer</u>: Our critical point dryer was purchased from Electron Microscopy Sciences, Inc. (Model # EMS 850) which is capable of thermoelectric heating and adiabatic cooling with a temperature control +5°C on cooling and +35°C on heating. It avoids excess pressures or temperatures and ensures that the critical point is obtained. This obviates the need to rely on pressure relief valves to control pressure during the heating cycle. It is designed for use with CO<sub>2</sub>, after first replacing any water in the specimen by a series of dehydration steps.

### E. Example Images Taken at NMMC

Below shows a variety of images taken by the table top SEM.

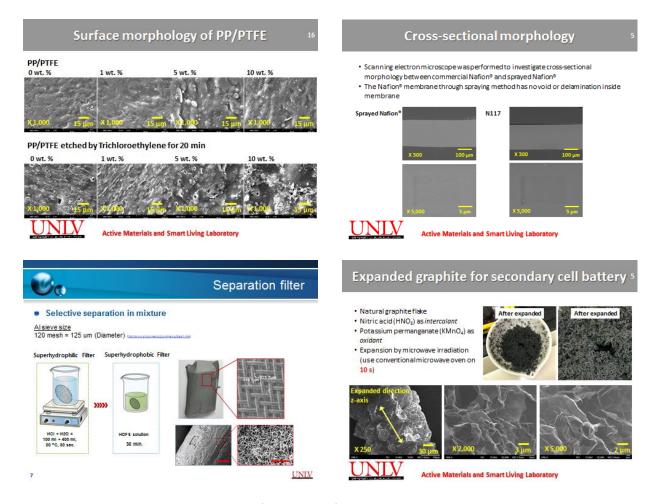


Figure 4. Various Images

## F. Work Plan

All the equipment was purchased and installed properly. They are operational.

## **G. Major Problem/Issues**

None.

# **H. Project-related Publications**

J.2 A New Ionic Polymer-Metal Composite Based on Nafion / poly(vinyl alcohol-co-ethylene)
Blends

T. Hwang, V. Palmre, J. Nam, D.-C. Lee, and K. J. Kim (submitted for review)

J.1 A Comprehensive Physics-Based Model Encompassing Variable Surface Resistance and Underlying Physics of Ionic Polymer-Metal Composite Actuators

Q. Shen, V. Palmre, T. Stalbaum, and K. J. Kim (submitted for review)

# Appendix – I



Figure Appendix-1. Exterior of Science and Engineering Building at UNLV



Figure Appendix-2. Science and Engineering Building Floor Plan for 2212 (The SEM and SECW are located in 2112A)